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(54) PROCESS FOR OBTAINING A PROTEIN CONCENTRATE FROM WHEY

(71) I, JOSEF ANTON-MEGGLE, a German citizen, trading as MOLKEREI J. A. MEGGLE MILCHINDUSTRIE, of 8094 Reitmehring, Germany, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with a process for obtaining a protein concentrate from whey, with the use of ultrafiltration.

From published Dutch Patent Specification No. 70 10 656, it is known that milk and whey can be freed from low molecular weight components by ultrafiltration, with the formation of a concentrate of high molecular weight milk proteins. An advantage of this process is that the protein or protein concentrate gives the proteins in their original form, which is not so in the case of other processes for obtaining proteins from milk and whey. A disadvantage of this process, which preserves the proteins is, however, that whey in particular is a material which is of very considerably varying bacteriological quality and that the process conditions offer sufficient scope for bacteriological activity so that the end product cannot be of satisfactory bacteriological quality when a satisfactory starting material is not available. However, a satisfactory starting material can only be ensured by heating the whey beyond the extend of conventional pasteurising. However, this impairs the consistency of the protein, i.e. the protein begins to flocculate out. Whey denatured to this extent cannot be used or can only be used with great difficulty in an ultrafiltration process.

Therefore, it is an object of the present invention to overcome these difficulties and to provide a process for concentrating whey protein to give a bacteriologically satisfactory concentrate which also has an acceptable flavour or taste.

If an attempt is made to use previously pasteurised whey in the known process then this does not give the desired result because during ultrafiltration, it is practically impossible to prevent fresh infection. The concentrated product of the ultrafiltration also cannot be heat-sterilised because a considerable part of the protein thereby precipitates out, which impairs heat transfer in the heating plant and can even block up the plant. Furthermore, overheating and protein losses can occur.

Surprisingly, these difficulties can be overcome by the process according to the present invention, wherein whey is adjusted to a pH value below the isoelectric point of the whey protein, preferably to pH 2.7 to 3.3, thereafter subjected to ultrafiltration and heated to ensure microbial destruction before and/or after the ultrafiltration, the product obtained then, if desired, being neutralised and optionally dried.

Heating to ensure microbial destruction can be carried out under the conditions of a conventional pasteurisation or at higher temperatures before and/or after the ultrafiltration. In principle, at least one heating step is necessary for bacteriological reasons. It is especially surprising that, even after the ultrafiltration, the already concentrated product can be heated to the usual pasteurisation temperatures without the abovementioned disadvantages arising. It is, therefore, possible to work up a whey protein concentrate, prepared according to the process of the present invention, without difficulty with the use of indirect heating systems. This is because no significant turbidity occurs due to flocculating out of protein.

Furthermore, we have, surprisingly, ascertained that in the case of the above-given pH values, a practically complete destruction of spore formers and of their spores takes place at temperatures above 85°C., especially at temperatures between 90 and 100°C. Thus, the process according to the present invention permits the production of quasi sterile whey protein products.

The process according to the present invention can be applied to wheys from rennet cheese making, whey from curd cheese making, whey from rennet casein and from curd casein production, partially desaccharified whey, as well as partially desaccharified and

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partially desalinated whey and mixtures of various wheys and whey derivatives. Wheys of the most varied qualities can be used in natural form or after preconcentration, a preferred concentration range being 6 to 15% of dry materials in the starting material. Normally, non-concentrated whey usually has a dry material content of about 6%. Starting concentrates with a dry material content of more than 15% can also be used. However, in this case, care is to be taken that the concentrate is not too viscous in order that the ultrafiltration is not made too difficult, which could impair the economic success of the process. Therefore, a dry material content of 15% and over will normally only be considered when the process according to the present invention is carried out in several stages, i.e. the whey protein, which has already been concentrated by ultrafiltration, is subjected to at least one further ultrafiltration.

An essential feature of the process according to the present invention is the adjustment of a pH value to below the isoelectric point of the protein and preferably to a pH value within the given range. It is possible to go below the given pH range when care is taken that at such a low pH value, no hydrolytic fission of the protein takes place which could bring about an impairment of the taste. However, at pH values below 2.7, heating before the ultrafiltration is expediently omitted in order to be certain to exclude a hydrolytic fission. After the ultrafiltration, the pH value of the product is then expediently increased to the preferred range by partial neutralisation and heating is then carried out within this range. Products with pH values below 2.7 are also only of limited use so that, from this aspect, too, a neutralisation, i.e. increasing of the pH value above the isoelectric point or increasing the pH value above 2.7 is expedient.

The adjustment of the desired pH value can be carried out in any desired manner by known methods, for example, by the addition of acids, by the removal of cations with the use of ion exchangers or by electrodialysis. It is also possible to carry out the acidification with strongly acidifying microorganisms, for example, of the *Lactobacillus helveticus* type.

The heating of the starting material takes place at normal pasteurisation temperatures, preferably at temperatures below 75°C., for example between 70 and 75°C. It is important that the heating conditions are such that the whey protein is not or is only slightly damaged, which is ensured under the given conditions.

In the case of heating the concentrate, it is possible to use not only normal pasteurisation temperatures but also considerably higher temperatures. Therefore, the concentrate is preferably heated to temperatures of 85°C. or above. If temperatures are used which are substantially above 100°C., then it is expedient to use apparatus which ensures that the concentrate does not form a thin film on the surface of the heater when indirect heating is employed, because such a film makes difficult the transmission of heat and the operation of the plant. If, however, heating is carried out directly, for example by blowing in steam, then very high temperatures can be achieved and thus a really outstanding microbiological purity is obtained. Conventional heating devices can be used not only for heating the starting materials but also for heating the concentrates. Plate heaters are especially preferred for heating the starting material. However, plate heaters can also be used for heating the concentrate because the protein remains completely in solution under the conditions according to the present invention. The selected heating conditions depend upon the desired degree of protein denaturing and of sterility.

Ultrafiltration itself is a known process and does not need to be described here in detail. It is preferred to select a filtering medium having a pore size which, on the one hand, substantially retains the protein and, on the other hand, allows low molecular weight materials, such as salts and lactose, to pass into the filtrate as far as possible. Preferably, not more than 0.1 to 0.15% protein should be contained in the filtrate. The expedient upper concentration limit for the product of the ultrafiltration is about 25% dry mass. In the case of still higher dry mass contents, disturbances of the ultrafiltration can occur. A dry mass in the region of the given upper limit is expediently not to be sought in a single passage through the ultrafiltration but rather by at least two passages, concentration first being carried out to a somewhat lower dry mass content and only then in the second or subsequent step to the final dry mass content. Such a method of working has the advantage that the lactose content of the end product is much more considerably reduced than when the final content of the dry mass is achieved by only a single ultrafiltration.

Neutralisation of the ultrafiltration concentrate is carried out if, for the intended use, a higher pH value is desired. Neutralisation can be carried out in known manner, for example, by the addition of alkalis, for example of citrates, carbonates or hydroxides of alkali metals or of alkaline earth metals, care being taken that a localised alkalisation is avoided, for example, by an appropriately measured addition of the alkali and rapid mixing. Neutralisation of the concentrate can be especially of interest when, in the end product, there is desired a composition of

ions which differs from the usual one, for example, an increasing or decreasing of the calcium, potassium or sodium content. In this way, it is possible to "tailor make" the mineral composition of the product.

The product of the process according to the present invention can be used directly as a highly viscous, liquid concentrate or it can be dried. Preferred drying processes are, for example, spray drying and freeze drying. We have ascertained that the special properties of the whey protein concentrate obtained by the process according to the present invention are retained under the conditions of spray drying.

The process according to the present invention can be used for a continuous carrying out of the ultrafiltration, which is especially economical. Hitherto, this was not possible because it meant that the ultrafiltration plant was constantly in operation and, over very long periods of operation, there were present very favourable growth conditions for microorganisms when, as hitherto, the operation was carried out at a neutral pH value.

The product obtained by the process according to the present invention has a number of especially advantageous properties which ensure a multiplicity of possible uses. The opinion was previously held that whey protein products show flavour disadvantages in the course of a process which is carried out at low pH values. Surprisingly, the process according to the present invention has shown this to be untrue. On the contrary, we have ascertained that, after passing through a pH value range below the isoelectric point, i.e. at about pH 3, no irreversible aroma damage occurs, which proves itself in that the products produced by the process according to the present invention, upon again adjusting to higher pH values, have a taste which is the same as that of natural milk products. Furthermore, the product of the process according to the present invention is precipitatable to a lesser extent than the products treated according to the known processes. From this, it follows that the protein is denatured to a lesser extent and thus has a higher content of native protein than is the case after heating at pH values around the isoelectric point or above. This increases the nutritional-physiological value of the product so that it is especially suitable for the preparation of dietetic foodstuffs. Furthermore, the process according to the present invention permits the production of germ-free, sterile products which was previously not possible without impairment of taste.

The process itself also has considerable advantages. In particular, the energy requirements are reduced because the quantities to be heated are smaller, it is possible to heat with simple plate heaters without blockage occurring or without precipitation of protein in the concentrate.

The dry mass passage through the membranes in the case of the ultrafiltration is high and reaches substantially the maximum passthrough values which are achieved at pH values of about 6 and is clearly above the values which are achieved at about the isoelectric point or at pH values in the neutral or weakly alkaline range.

The following Examples are given for the purpose of illustrating the present invention:

Example 1.

1000 litres of fresh whey with a content of 4.8% lactose, 0.8% protein and 0.5% minerals are divided up into two approximately equal parts and one part is passed over a cation exchanger. The whey thus obtained has a pH value between 1.5 and 1.8. It is mixed with the other, untreated half of the whey. There is thus obtained a mixture with a pH value of between 2.8 and 3.2. The mixture contains 4.8% lactose, 0.75% protein and 0.35% minerals.

Subsequently, the mixture is pasteurised under the conditions normally used in dairies and then subjected to ultrafiltration. There are obtained 900 litres of permeate with a content of 4.6% lactose, 0.2% protein and 0.3% minerals, as well as 100 litres of concentrate with a content of 6.6% lactose, 5.7% protein and 0.8% minerals (dry mass 13.1%).

The concentrate thus obtained is further concentrated in a down-flowing evaporator to a dry mass content of 30% and then spray dried. Before spray drying, the concentrate can be neutralised to various pH values.

Example 2.

This Example illustrates the dry mass output at various pH values.

In ultrafiltration technology, the throughput is given as permeate dry mass/hour/m^2.

The process of Example 1 is repeated while maintaining a pH value of 3. The throughput is 1.1 kg./hr./m^2.

For the purpose of comparison, the following experiments are carried out:

The experiment is repeated under the same conditions except that the pH value is at the isoelectric point. The throughput is 0.64 kg./hr./m^2.

The process is again repeated with adjustment of the pH value to 6. The throughput is 1.28 kg./hr./m^2.

At higher pH values (above 6.5), the throughput falls still further and, at neutral pH, has the same value as at pH 3.

The concentrates obtained at pH 6 or at the isoelectric point give, upon attempting to concentrate them to 30% dry mass content, a marked formation of precipitate in

the heater. The product spray dried at these pH values was unsatisfactory from the point of view of taste.

Example 3.

For further concentration with regard to the protein content, the concentrate obtained according to Example 1 is diluted with an equal amount of water to a lactose content of 3.3%, a protein content of 2.85% and a mineral content of 0.4%. This mixture (100 litres) is again subjected to ultrafiltration with the formation of 75 litres of permeate with a content of 3.2 lactose, 0.05% protein and 0.3% minerals, as well as of 25 litres of concentrate with a content of 3.6% lactose, 11.25% protein and 0.7% minerals. This concentrate is further concentrated in the manner described in Example 1 to a dry mass content of 20% and then spray dried.

Example 4.

Removal of bacteria from the concentrate.

The concentrate obtained according to Example 1 is preheated to 80°C. in a plate heater and then, by means of steam injection, heated further, with strong stirring, to 90 to 95°C. The heated product shows no coagulation phenomena and is an opaque, flowable mass. After a heating period of about 10 minutes, it is spray dried to give a powder.

Bacteriological quality:

Less than 10 bacteria or moulds or yeasts per gram; less than 10 aerobic spore formers per gram; test for anaerobic spores (Weinzirl test) negative after 5 days.

Example 5.

1000 kg. sour whey from the production of curds with a content of 4.3% lactose, 0.75% protein, 0.65% minerals and 0.5% lactic acid are passed over a cation exchanger. The product thus obtained has a pH value between 1.5 and 1.8. The partially desalinated whey thus obtained contains 4.3% lactose, 0.7% protein, 0.35% minerals and 0.5% lactic acid. This whey is mixed with about 300 kg. sour whey concentrate (21% lactose, 3.7% protein, 3.3% minerals and 2.5% lactic acid), a whey being obtained with a dry mass content of 12%, a content of 8.1% lactose, 1.4% protein, 1.03% minerals and 0.65% lactic acid and a pH value between 2.8 and 3.1. Ultrafiltration of this whey gives 1170 kg. of permeate with a content of 7.8% lactose, 0.4% protein, 0.9% minerals and 0.9% lactic acid, as well as 130 kg. of concentrate with a content of 10.8% lactose, 10.4% protein, 2.2% minerals and 1.2% lactic acid. The concentrate thus obtained is worked up in the manner described in Example 1 and spray dried.

WHAT I CLAIM IS:—

1. Process for the production of a whey protein concentrate by ultrafiltration, wherein whey is adjusted to a pH value below the isoelectric point of the whey protein, thereafter subjected to ultrafiltration and heated to ensure microbial destruction before and/or after the ultrafiltration.

2. Process according to claim 1, wherein the whey is adjusted to a pH value of 2.7 to 3.3.

3. Process according to claim 1 or 2, wherein the pH value of the whey is adjusted by electrodialysis or by removal of cations with the use of ion exchangers or by the addition of an acid or with the use of strongly acidifying microorganisms.

4. Process according to any of the preceding claims, wherein, before the ultrafiltration, the whey is heated to a temperature between 70 and 75°C.

5. Process according to any of the preceding claims, wherein, after the ultrafiltration, the concentrate obtained is heated to a temperature above 85°C.

6. Process according to any of the preceding claims, wherein the whey used has a dry mass content between 6 and 15%.

7. Process according to any of the preceding claims, wherein the ultrafiltration concentrate obtained is diluted with water and again subjected to ultrafiltration.

8. Process according to any of the preceding claims, wherein the ultrafiltration is carried out continuously.

9. Process according to any of the preceding claims, wherein the concentrate obtained is neutralised.

10. Process according to claim 9, wherein the neutralisation is carried out with an alkali metal or alkaline earth metal citrate, carbonate or hydroxide.

11. Process according to any of the preceding claims, wherein the concentrate is dried.

12. Process according to claim 11, wherein drying is carried out by spray drying or freeze drying.

13. Process according to claim 1 for the production of a whey protein concentrate, substantially as hereinbefore described and exemplified.

14. Whey protein concentrates, whenever produced by the process according to any of claims 1-13.

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